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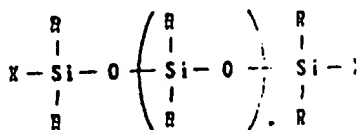
## SPECIFICATION

### 1. Title of the Invention

Makeup cosmetic

### 2. Claims

1. A makeup cosmetic, characterized by containing an organic silicone resin obtained by condensing (a) one or more types of polydiorganosiloxane substantially as



shown by the general formula

(where R are the same or different substituted or unsubstituted monovalent hydrocarbon groups, X is a hydroxyl group or hydrolyzable group, and n is an integer of 10-20,000) and (b) one or more types of polyorganosiloxane that is a copolymer of siloxane units shown by the formula  $\text{R}_3\text{SiO}_{0.5}$  and siloxane units shown by the formula  $\text{SiO}_2$  (where R are the same or

different substituted or unsubstituted monovalent hydrocarbon groups) and has at least one hydroxyl group or hydrolyzable group bonded to a silicon atom in each molecule.

2. The makeup cosmetic according to Claim 1, wherein the cosmetic contains a low-boiling solvent.
3. The makeup cosmetic according to Claim 2, wherein the low-boiling solvent is at least one solvent selected from among low-boiling silicones, low-boiling isoparaffin-based hydrocarbons having 1-30 carbon atoms, and chlorofluoro-substituted hydrocarbons.

### 3. Detailed Description of the Invention (Field of Industrial Utilization)

The present invention relates to a makeup cosmetic, and more particularly to a durable, stable makeup cosmetic with excellent water resistance, perspiration resistance, and oil resistance. As used herein, the term "makeup cosmetic" includes undercoating cosmetics used as an undercoating in addition to ordinary makeup cosmetics.

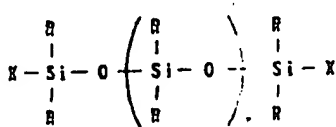
(Technological Background of the Invention and Problems Thereof)

There are various type of makeup, such as solid foundation,

solid eye shadow, oily foundation, and lipstick. There are also emulsified products such as emulsified foundation. All of them are characterized by containing large amounts of inorganic pigments such as kaolin, iron oxide, titanium oxide, titanium, and micaceous pearl pigment; and organic pigments such as nylon, cellulose, and tar pigments.

These makeup cosmetics tend to run due to oil and perspiration on the skin or to the oil contents of the cosmetic. Frequent perspiration in the summer in particular makes running of the makeup a serious problem that requires improvement. Recent greater participation in sports as a hobby has also made it an everyday practice to wear makeup while playing sports. These makeup cosmetics, which are called sports makeup or summer makeup, must have greater than the usual water resistance, perspiration resistance, and oil resistance.

Undercoating makeup is used to improve the adhesion of makeup cosmetics and to produce a clean finish. However, few conventional makeup cosmetics are designed to provide durability.



Various attempts have been made to use polyorganosiloxanes to resolve such problems. However, combinations of linear polydiorganosiloxanes and siloxanes made from  $\text{R}_3\text{SiO}_{0.5}$  units and  $\text{SiO}_2$  units (R is a monovalent organic group) were used in these conventional makeup cosmetics. These cosmetics failed to yield adequate water repellency.

(Object of the Invention) The object of the present invention is to provide a long-lasting makeup cosmetic that has excellent water resistance, perspiration resistance, and oil resistance.

(Constitution of the Invention) As a result of research aimed at resolving the aforementioned problems, the inventors perfected the present invention upon discovering that the above-stated goal can be attained by the addition of a specific silicone resin. Specifically, the present invention is a makeup cosmetic characterized by containing an organic silicone resin obtained by condensing (a) one or more types of polydiorganosiloxane

(referred to as component (a) hereinafter) shown by the general formula (where R are the same or different substituted or unsubstituted monovalent hydrocarbon groups, X is a hydroxyl group or hydrolyzable group, and  $n$  is an integer of 10-20,000) and (b) one or more types of polyorganosiloxane (referred to as component

(b) hereinafter) that is a copolymer of siloxane units shown by the formula  $\text{R}_3\text{SiO}_{0.5}$  and siloxane units shown by the formula  $\text{SiO}_2$  (where R are the same or different substituted or unsubstituted monovalent hydrocarbon groups) and has at least one hydroxyl group or hydrolyzable group bonded to a silicon atom in each molecule.

The polydiorganosiloxane of (a) used in the present invention is essentially one that has hydroxyl groups or hydrolyzable groups at the two ends thereof. Examples of such hydrolyzable groups include condensation-reactive groups such as alkoxy groups, ketoxime groups, and aminoxy groups. Alkoxy groups such as methoxy, ethoxy, propoxy, and butoxy groups are common. These are essentially straight-chained, but may contain no more than 10% branching. The average degree of polymerization ( $n$ ) is 10-20,000, which corresponds to a molecular weight of approximately 700-1,500,000.

Component (b) that serves as the raw material of the organic silicone resin used in the present invention is well known to those skilled in the art. It is obtained by hydrolytic condensation of at least one type of material selected from water glass, orthoethyl silicate, and ethyl polysilicate (which constitute the  $\text{SiO}_2$  units), and at least one silane selected from trimethylchlorosilane, trimethylmethoxysilane, triphenylchlorosilane, phenyldimethylmethoxysilane, dimethylvinylchlorosilane, and dimethylvinylmethoxysilane (which constitute the  $\text{R}_3\text{SiO}_{0.5}$  units).

It is preferable to use a polyorganosiloxane whose molar ratio of siloxane units shown by the formula  $\text{R}_3\text{SiO}_{0.5}$  and siloxane units shown by the formula  $\text{SiO}_2$  is 0.5-1.0, whose average molecular weight is about 2000-3000, and which is a copolymer that has at least one hydroxyl group or hydrolyzable group bonded to a silicon atom in each molecule.

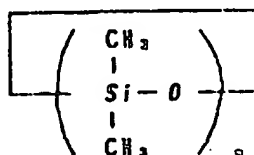
The organic silicone resin of the present invention is obtained by partial condensation of component (a) and component (b). The ratio of component (a) and component (b)

in this organic silicone resin is preferably from 80:20 to 20:80. This partial condensation reaction is conducted without a catalyst or in the presence of a basic catalyst. Examples of such basic catalysts include metal hydroxides such as sodium hydroxide and lithium hydroxide, aliphatic amines such as trimethylamine and diethylamine, aromatic amines such as aniline and methyl aniline, and quaternary ammonium compounds such as organoammonium hydroxide.

The organic silicone resin of the present invention is added in an amount of 0.1-50% by weight in relation to the total weight of the makeup cosmetic. The effect is inadequate if the amount is less than 0.1% by weight, and the product becomes difficult to dissolve in a low-boiling solvent if the amount exceeds 50% by weight.

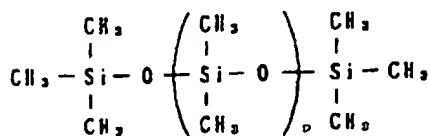
The makeup cosmetic of the present invention usually contains the solvents described below. Low-boiling solvents that evaporate at room temperature may be used. Low-boiling linear silicones, cyclic silicones, low-boiling isoparaffin-based hydrocarbons, chlorofluoro-substituted hydrocarbons, and the like are preferred as the low-boiling solvents.

Low-boiling silicones are represented by the general formula shown below. Specific examples include hexamethyldisiloxane, octamethyltrisiloxane, decamethyltetrasiloxane, and dodecamethylpentasiloxane.



(where  $p$  is an integer of 0-5).

Cyclic siloxanes are represented by the general formula shown below. Specific examples include octamethylcyclotetrasiloxane, decamethylcyclopentasiloxane, and dodecamethylcyclohexasiloxane.



(where  $q$  is an integer of 3-7).

Examples of low-boiling isoparaffin-based hydrocarbons are isoparaffin-based hydrocarbons having 1-30 carbon atoms with a boiling point in the 50-300°C range at normal temperature.

Examples of the chlorofluoro-substituted hydrocarbons include trichlorotrifluoroethane and tetrachlorodifluoroethane. The aforementioned low-boiling solvents can be used singly or as any combination of two or more solvents. The total amount added should be no more than 99% by weight, and preferably 10-90% by weight, in relation to the total weight of the hair cosmetic.

Low-boiling alcohols such as ethanol and IPA may also be added besides the aforementioned components. A silicone derivative may also be added as an ingredient in accordance with the present invention. For example, it is possible to add one or more arbitrary components selected from linear dimethylsilicone oil, methylphenylsilicone oil, polyether-modified silicone oil, amino-modified silicone oil, epoxy-modified silicone oil, and fluorine-modified silicone oil.

These silicone derivatives are effective for modifying the organic silicone resin film. Adding a low-molecular-weight plasticizer is known to improve resin films, but the conventional plasticizers comprising phthalic acid derivatives and higher alcohol polyoxyethylene ethers are inappropriate from the standpoint of compatibility. In this regard, silicone derivatives have good compatibility with the organic silicone resin. They are effective in plasticizing the resin film, enhance adhesiveness to the hair, and can yield a perfect film that remains free from stickiness without losing its smoothness.

The amount in which these silicone derivatives are added is no more than 99% by weight in relation to the total weight of the hair cosmetic. Adding more than 99% by weight is undesirable because the organic silicone resin fails to form a film in this case.

The hair cosmetic of the present invention can be produced in any form. It can be a solubilized system, emulsified system, dispersed powder system, oil-water two-layered system, or oil-water-powder three-layered system.

The oil phase that contains the organic silicone resin is commonly emulsified by a nonionic surfactant, cationic surfactant, anionic surfactant, or a mixture thereof in the case of an emulsified system. It is preferable, however, to adopt a method in which a mixture of a surfactant and a water-soluble polyhydric alcohol is prepared in advance, and the product is then mixed with an oil phase, yielding a composition.

The water-soluble polyhydric alcohol has at least two hydroxyl groups per molecule. Specific examples include ethylene glycol, propylene glycol, 1,3-butylene glycol, 1,4-butylene glycol, dipropylene glycol, glycerol, polyglycerol (such as diglycerol, triglycerol, tetraglycerol, and decaglycerol), glucose, maltitol, sucrose, fructose xylitol, erythritol, and amylolytic sugar reduced alcohol. Any one or more of the above may be selected and used.

The following are examples of oils that can be used in addition to the organic silicone resin of the present invention as the oil phase of an emulsified cosmetic.

Specific examples include oils commonly used in cosmetics, such as olive oil, coconut oil, safflower oil, castor oil, cottonseed oil, and other fats and oils; lanolin, jojoba oil, carnauba wax, and other wax oils; liquid paraffin, squalene, petrolatum, volatile isoparaffin, and other hydrocarbon oils; fatty acids; alcohols; cetyl octanoate, isopropyl myristate, and other ester oils; dimethylpolysiloxane, methylphenylpolysiloxane, and other silicone oils; and silicone resins.

The aforementioned ingredients are commonly mixed in amounts such that the amount of surfactant is 0.5-10% by weight in relation to the total weight of the hair cosmetic, and the amount of the oil phase containing the organic silicone resin is 20-80% by weight. When an emulsified composition is obtained by preparing a mixture of surfactant and water-soluble polyhydric alcohol in advance and then mixing the product with the oil phase, the amount of surfactant is preferably 1-20% by weight, the amount of the oil phase containing the organic silicone resin is 10-70% by weight, and the amount of the water-soluble polyhydric alcohol is 5-30% by weight in relation to the total weight of the cosmetic, and about 50-90% by weight in relation to the surfactant.

The emulsified cosmetic may be either in the form of a water-in-oil emulsion or in the form of an oil-in-water

emulsion. However, these should be prepared in a manner that allows the product to preserve its volatility as a merit of the present invention.

Depending on the intended goal, the makeup cosmetic of the present invention may also contain, in addition to the aforementioned constituent components, fragrances as well as ultraviolet absorbers, antioxidants, preservatives, vitamins, hormones, and other chemical agents within the quantitative and qualitative ranges that do not compromise the effects of the present invention.

#### (Working Examples)

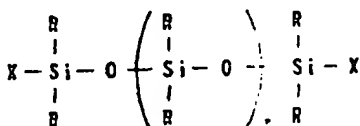
The present invention is explained in greater detail below through working examples. However, the present invention is not limited by these examples. All of the mixing amounts cited in the examples are indicated in percent by weight.

#### Reference example

50% toluene solutions of components (a) and (b) shown in Table 1 were mixed in amounts indicated as the weight ratios [(a)/(b)] of components (a) and (b), sodium hydroxide was then added in an amount of 0.002 part per 100 parts of component (a), and a condensation reaction was conducted under toluene refluxing while the system was heated and stirred, yielding organic silicone resins I-V.

Table 1

Organic silicone resin	(a)	(b)	(a)/(b)
I	R: methyl group X: hydroxyl group n: 7000	$(\text{CH}_3)_3\text{SiO}_{0.3}/\text{SiO}_2$ **0.8 Degree of polymerization: 2500	30/70
II	R: methyl group X: hydroxyl group n: 4000	$(\text{CH}_3)_3\text{SiO}_{0.3}/\text{SiO}_2$ **0.6 Degree of polymerization: 2000	45/55
III	R: phenyl group (10 mol%) Balance: methyl groups X: hydroxyl group n: 5000	$(\text{CH}_3)_3\text{SiO}_{0.3}/\text{SiO}_2$ **0.7 Degree of polymerization: 3000	20/80
IV	R: methyl group X: hydroxyl group n: 50	$(\text{CH}_3)_3\text{SiO}_{0.3}/\text{SiO}_2$ **0.8 Degree of polymerization: 2500	70/30
V	R: methyl group X: hydroxyl group n: 800	$(\text{CH}_3)_3\text{SiO}_{0.3}/\text{SiO}_2$ **0.7 Degree of polymerization: 2200	50/50



The following were used as polysiloxanes for comparison.

P1 = R and X in formula (a): methyl groups, n = 7000

P2 = 30/70 mixture of P1 and  $(\text{CH}_3)_3\text{SiO}_{0.3}/\text{SiO}_2 = 0.7$ , degree of polymerization 2500

Working example 1. Oily foundation

(1) Kaolin:	25.0%
(2) Titanium dioxide:	15.0%
(3) Red iron oxide:	3.0%
(4) Microcrystalline wax:	4.0%
(5) Liquid paraffin:	3.0%
(6) Sorbitan sesquiolate:	1.0%
(7) Decamethylcyclopentasiloxane:	39.0%
(8) Organic silicone resin (I):	8.0%
(9) Isopropyl palmitate:	2.0%
(10) Fragrance:	q.v.

Components (4) to (9) were stirred and dissolved at 75°C, and components (1) to (3) were then added and dispersed. The system was degassed, component (10) was added, and the product was packed into an appropriate container, yielding an oily foundation.

Working example 2. Liquid lipstick

(1) Dimethylsiloxane (1.5 cSt):	64.0%
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(2) Organic silicone resin (II):	20.0%
(3) Glyceryl triisostearate:	6.0%
(4) Red No. 226:	10.0%
(5) Fragrance:	q.v.

Components (1) and (2) were stirred and dissolved at 70°C, and a product obtained by separately treating components (3) and (4) with a roller was added and allowed to decompose. The system was degassed, and component (5) was added, yielding a liquid lipstick.

Working example 3. Mascara

(1) Light liquid isoparaffin:	55.0%
(2) Organic silicone resin (III):	30.0%
(3) Black iron oxide:	14.5%
(4) POE (20) sorbitan monolaurate:	0.5%
(5) Fragrance:	q.v.

Components (1) and (2) were stirred and dissolved at 40°C, and components (3) and (4) were then added and dispersed. The system was degassed, and component (5), yielding a mascara.

Working example 4. Cosmetic undercoating

(1) Kaolin:	10.0%
(2) Titanium dioxide:	5.0%
(3) Red iron oxide:	0.3%
(4) Yellow iron oxide:	0.2%
(5) Methyl phenyl polysiloxane (made by Toshiba Silicone, TSF437):	20.0%
(6) Squalene:	10.0%
(7) Solid paraffin:	5.0%
(8) Microcrystalline wax:	4.0%
(9) Sorbitan sesquiolate:	1.0%
(10) Organic silicone resin (IV):	2.0%
(11) Isopropyl myristate:	24.5%
(12) Fragrance:	q.v.

Components (1) to (4) were mixed and pulverized, a product obtained by separately mixing and dissolving components (5) to (11) at 80°C was stirred and mixed, the system was degassed, and component (12) was added, yielding a cosmetic undercoating.

Working example 5. Highlighter

(1) Octamethylcyclotetrasiloxane:	95.0%
(2) Organic silicone resin (V):	4.5%
(3) Titanium-mica-based pearl pigment:	0.5%

Components (1) and (2) were heated and dissolved, and components (3) and (4) were added and dispersed to obtain a highlighter.

Comparative example 1. Oily foundation

(1) Kaolin:	25.0%
(2) Titanium dioxide:	15.0%

(3) Red iron oxide:	3.0%
(4) Microcrystalline wax:	4.0%
(5) Liquid paraffin:	3.0%
(6) Sorbitan sesquioleate:	1.0%
(7) Decamethylcyclopentasiloxane:	39.0%
(8) Polysiloxane: P1:	8.0%
(9) Isopropyl palmitate:	2.0%
(10) Fragrance:	q.v.

Components (4) to (9) were stirred and dissolved at 75°C, and components (1) to (3) were then added and dispersed. The system was degassed, component (10) was added, and the product was packed in an appropriate container, yielding an oily foundation.

#### Comparative example 2. Liquid lipstick

(1) Dimethylsiloxane (1.5 cSt):	64.0%
(2) Polysiloxane, P2:	20.0%
(3) Glyceryl triisostearate:	6.0%
(4) Red No. 226:	10.0%
(5) Fragrance:	q.v.

Components (1) and (2) were stirred and dissolved at 70°C, and a product obtained by separately treating components (3) and (4) with a roller was added and allowed to decompose. The system was degassed, and component (5) was added, yielding liquid lipstick.

The performance of the makeup cosmetics obtained in working examples 1-5 and comparative examples 1 and 2 was evaluated by the following test.

1 g each of the makeup cosmetics of working examples 1-5 and comparative examples 1 and 2 was applied to the arm and dried. Next, filter paper that had been moistened by water or squalene was pressed to the area where the cosmetic had been applied, and moved back and forth 10 times. After the back and forth movement had been completed, the amount of sample that had transferred to the filter paper was evaluated visually on the basis of color intensity. Evaluation was performed according to the following criteria by a panel of 10 women, and mean values were determined. The results are shown in Table 2.

- 1 ... No transfer at all
- 2 ... Slight transfer
- 3 ... Considerable transfer

Table 2

	Water	Squalene
Working example 1	1.1	1.1
Working example 2	1.2	1.3
Working example 3	1.1	1.3
Working example 4	1.0	1.1
Working example 5	1.0	1.0
Comparative example 1	2.4	2.8
Comparative example 2	1.9	2.2

#### (Merits of the Invention)

The makeup cosmetic of the present invention contains a specific organic silicone resin, and hence has excellent water resistance, perspiration resistance, oil resistance, and durability. The cosmetic is also easy to stretch and has a refreshing feel upon application.

Agent: Hajime Furutani, Patent Attorney (and three others)